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This is a new 3-year program to build on the success of the previously funded AFOST Contract FA9550-04-C-0020. In the last year of this program, we made major progress in both objectives of the program. We fabricated films of different thickness by our standard MOCVD processes and worked with our collaborators (ORNL, LANL, FSU,) to understand the microstructural reasons for the Ic performance using a variety of advanced characterization tools. We then modified our MOCVD process using a multipass technique to improve Ic performance in thick films. We modified rare-earth composition to improve critical current performance. This report addresses progress made in the first 3 months of the new program.

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**Final Report to AFOSR**

**Contract # FA9550-04-C-0020**

**September 2007**

by

**V. Selvamanickam, Program Manager**

**IGC-SuperPower**

**450 Duane Ave.**

**Schenectady, NY 12304**

## Objectives :

The objective of this program is as follows :

- *Process solutions to achieve high critical current in thick-film HTS 2G wires, and*
- *Development of conductor with improved magnetic field performance.*

## Status of Effort :

This is a new 3-year program to build on the success of the previously funded AFOST Contract FA9550-04-C-0020. In the last year of this program, we made major progress in both objectives of the program. We fabricated films of different thickness by our standard MOCVD processes and worked with our collaborators (ORNL, LANL, FSU,) to understand the microstructural reasons for the Ic performance using a variety of advanced characterization tools. We then modified our MOCVD process using a multipass technique to improve Ic performance in thick films. We modified rare-earth composition to improve critical current performance. *This report addresses progress made in the first 3 months of the new program.*

## Experimental :

MOCVD of YBCO was conducted in a custom-built facility at SuperPower described in previous Progress reports. The surface morphology of the YBCO films was examined by Field Emission Scanning Electron Microscope (FESEM) followed by compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS). In addition we also analyze bulk composition by Inductively Coupled Plasma (ICP) spectroscopy and elemental depth profiling by Glow Discharge Optical Emission Spectroscopy. Film cross sections were made with Focussed Ion Beam Milling (FIB). The texture of the films was analyzed by XRD including polefigure measurements. The thickness of the films was measured by surface profilometry.

## Accomplishments/New Findings :

In our Final Report for Contract FA9550-04-C-0020 that ended in May 2006, we reported improvements in thickness influence on Ic of MOCVD-based 2G wires. The small amount of CuO phases to the film surface. With such an enhanced microstructure, we more than doubled the Jc of our MOCVD-grown films from 2 – 2.5 MA/cm$^2$ to 4 – 5 MA/cm$^2$. Next, we employed a multipass approach to grown thicker films where each pass was a distinct MOCVD process run, and the subsequent layer was deposited atop the film processed in the previous pass. By this approach, we could modify the process conditions as needed in each pass. The thickness of film deposited in each pass was approximately 0.7 micron. Hence, a 2.1 micron film was produced in 3 passes. In a 2.1 micron thick film, we achieved a critical current of 557 A/cm corresponding to a Jc of 2.65 MA/cm$^2$. A summary of 2006 accomplishments is shown in Figure 1.

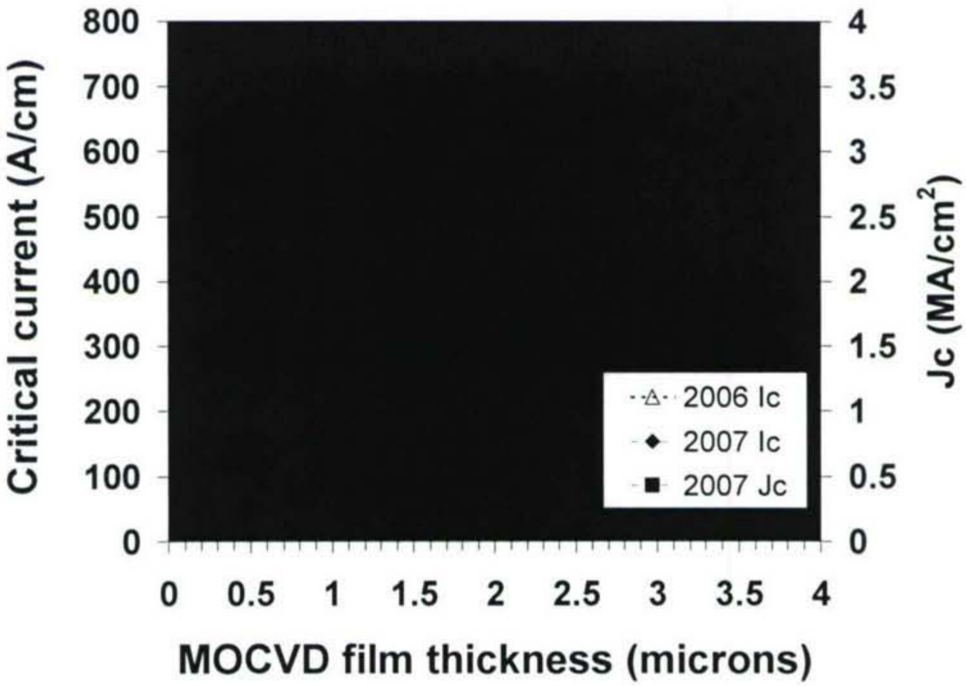


**Figure 1.** Critical currents and critical current densities of 2G conductor fabricated by MOCVD with different YBCO layer thickness in 2006 and 2007.

It can be seen from Figure 1 that in 2006, the Ic did not increase when the film thickness was increased from 2.1 to 3.5 microns. In this year, we examined the microstructural causes for this lack of Ic improvement and modified the MOCVD process to achieve higher currents, up to 720 A/cm in a 3.5 micron film corresponding to a Jc of 2 MA/cm$^2$. This critical current was measured over the entire width of the tape of 12 mm without patterning using continuous dc current (not a pulsed current) and a I-V curve obtained from the measurement is shown in Figure 2.

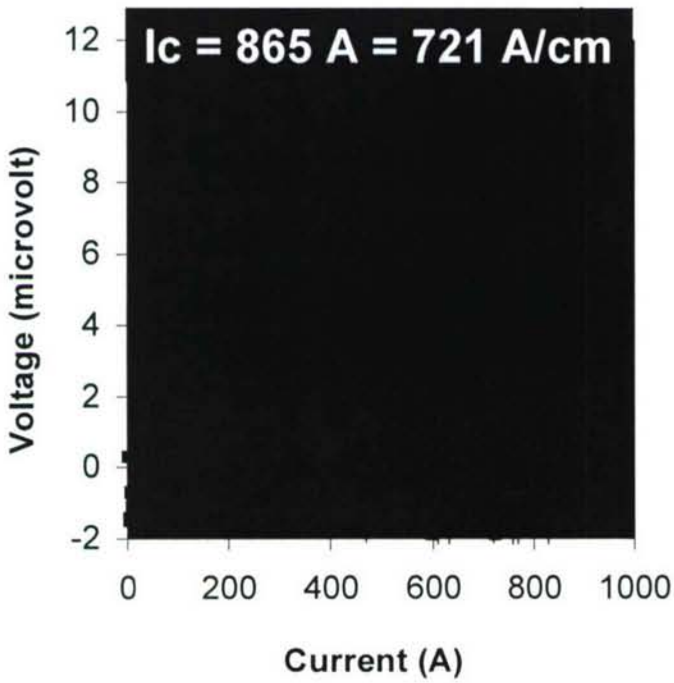


**Figure 1.** I-V curve obtained from measurement on a 3.5 micron thick HTS film fabricated by MOCVD. The measurement was done over the entire tape width of 12 mm without patterning.

This year, we also worked with FSU to characterize in detail the 2.1 micron film with a Ic of about 600 A/cm. Figure 3 shows Jc values obtained over a temperature range of 45 to 82 K and magnetic field up to 9 T. Detailed TEM examination was conducted on this sample to determine the source of flux pinning. Micrographs obtained from this examination are shown in Figure 4.

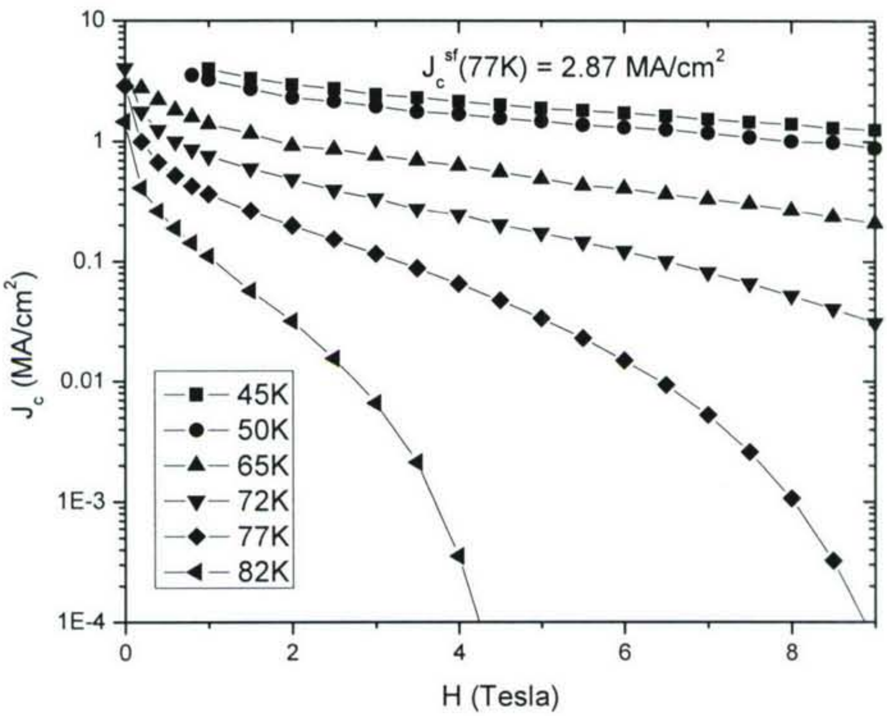


**Figure 3.** Critical current densities of 2G conductor with 2.1 micron thick HTS film fabricated by MOCVD measured at various fields and temperatures..

Plan view TEM images in Figure 4 show Sm-rich $Y_2O_3$ nano precipitates with average spacing of ~ 15 to 20 nm. The precipitates are seen to be dispersed in two major orientations. Cross-section TEM images show that the nano precipitates cluster on the stacking faults. HREM x-section images indicate the size of the nano-precipitates varies ~4 x 6 nm to ~10 x 20 nm with an average spacing of ~ 10 nm. Black arrows indicate stacking faults.

Clearly, our Jc MOCVD films consists of uniformly distributed, nanometer sized Sm-rich $Y_2O_3$ precipitates which could be the contributing microstructural factor for high Jc.

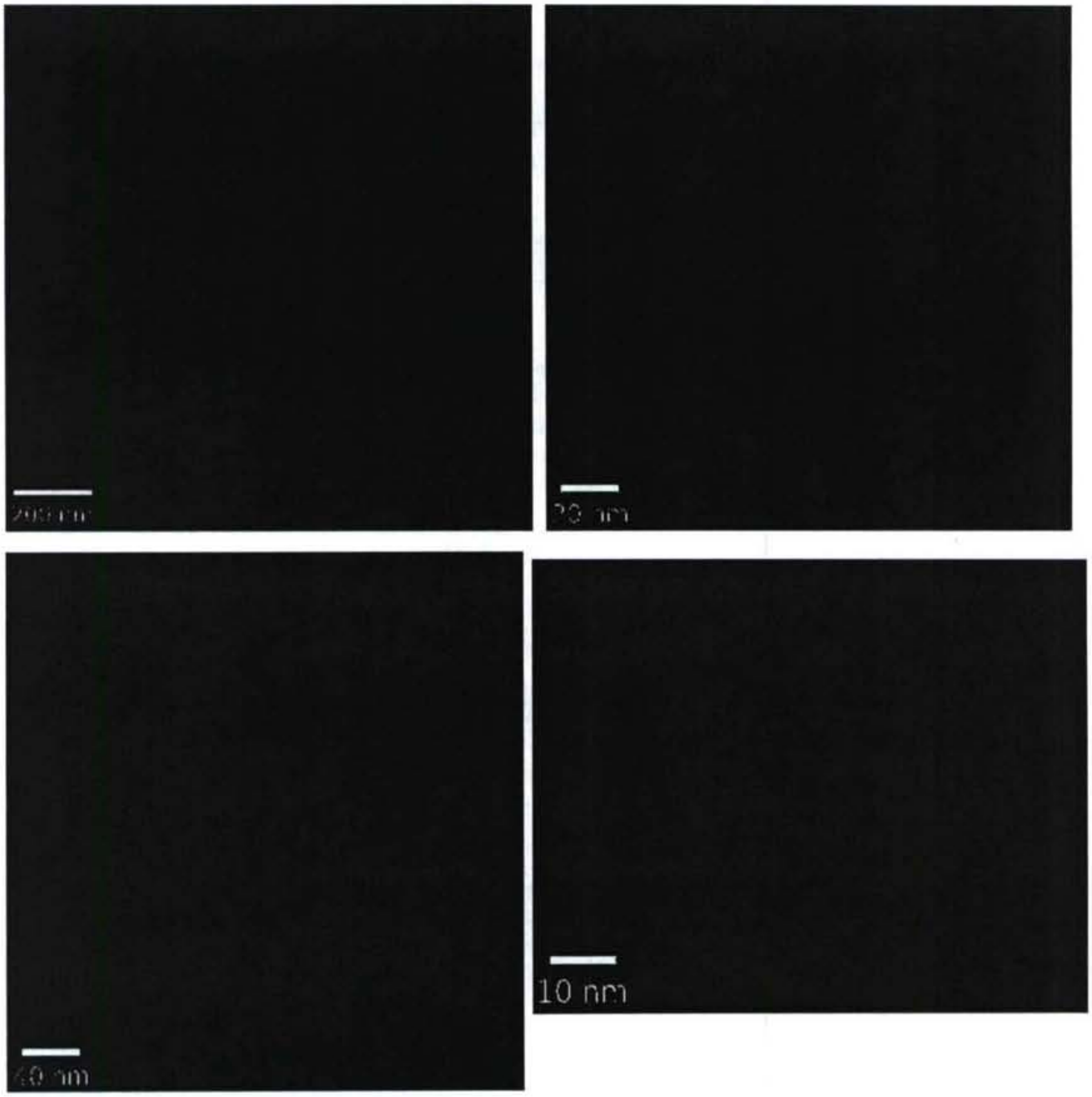


**Figure 4.** Plan view, cross section and High Resolution TEM images from a 2.1 micron thick HTS film grown by MOCVD.

Next, we scaled up the thick film process to long lengths. Figure 5 shows a I-V curve obtained from a meter-long MOCVD tape with a film thickness of 2.8 microns. At a voltage criterion of 0.27 microvolt/cm, we measured a critical current of 595 A/cm over the entire meter.

Figure 6 displays critical current data measured over a 10 m MOCVD tape with a film thickness of 2.8 microns. A critical current of 486 A/cm was measured over the 10 m.

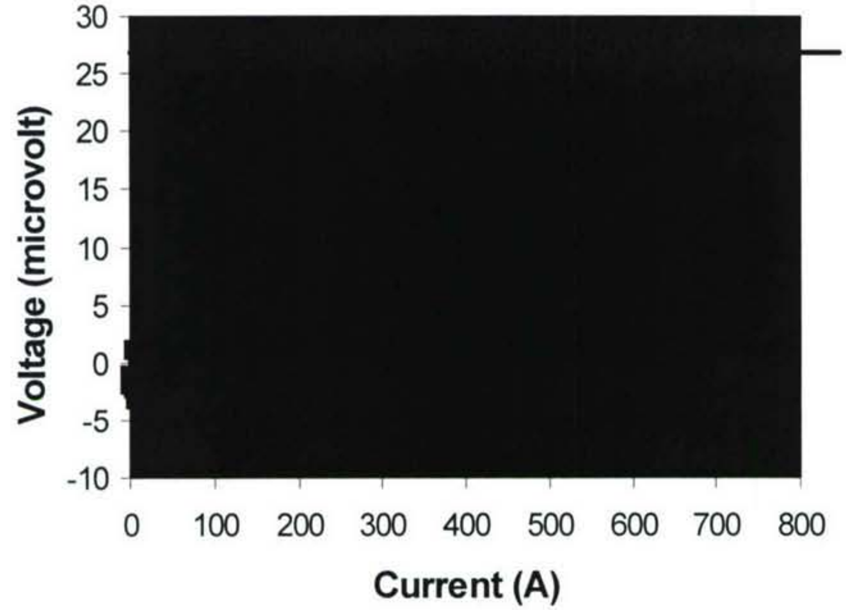


**Figure 5.** I-V curve obtained from a meter-long 2G wire with 2.8 micron HTS layer thickness. An end-to-end Ic of 595 A/cm was measured.

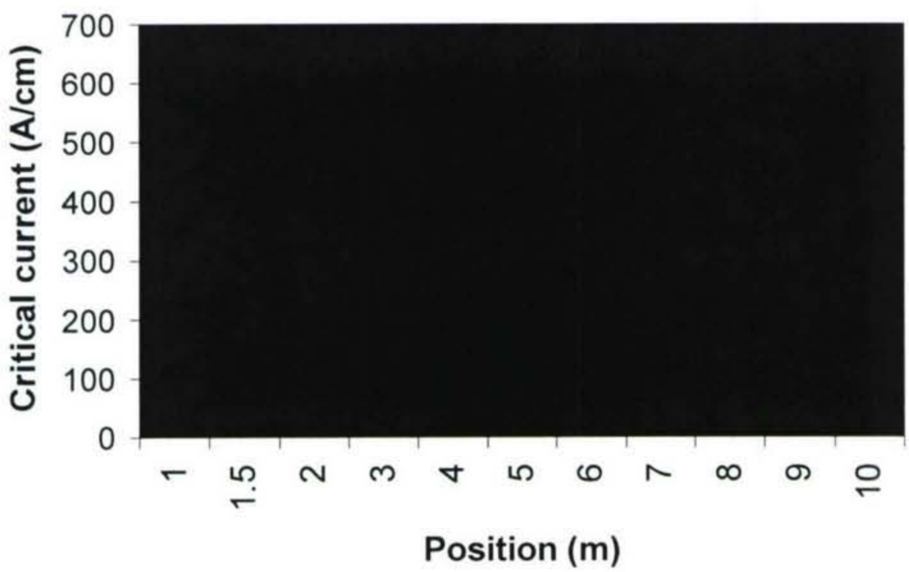


**Figure 5.** I-V curve obtained from a 10 m-long 2G wire with 2.8 micron HTS layer thickness. An end-to-end Ic of 486 A/cm was measured.

In an effort to further understand limiting factors for Jc in our thick films, we worked with LANL where microstructural examination was conducted on tapes after each of 4 passes. Figure 7 exhibits a cross sectional TEM image from a 2.8 micron MOCVD film. In addition to the vertical and horizontal defect structure which was previously characterized to be Y and Y-Cu-rich precipitates, a number of a-axis grain growths can also be seen in the top part of the film.

Figure 8 shows top surface SEM microstructure obtained from films deposited after 1, 2, 3, and 4 passes. It is clearly seen from this figure that the number of a-axis grains increase with the number of passes and could be a limiting factor for the Jc of thick films.

**Figure 7.** Cross section TEM image from a 2.8 micron thick HTS film grown by MOCVD.

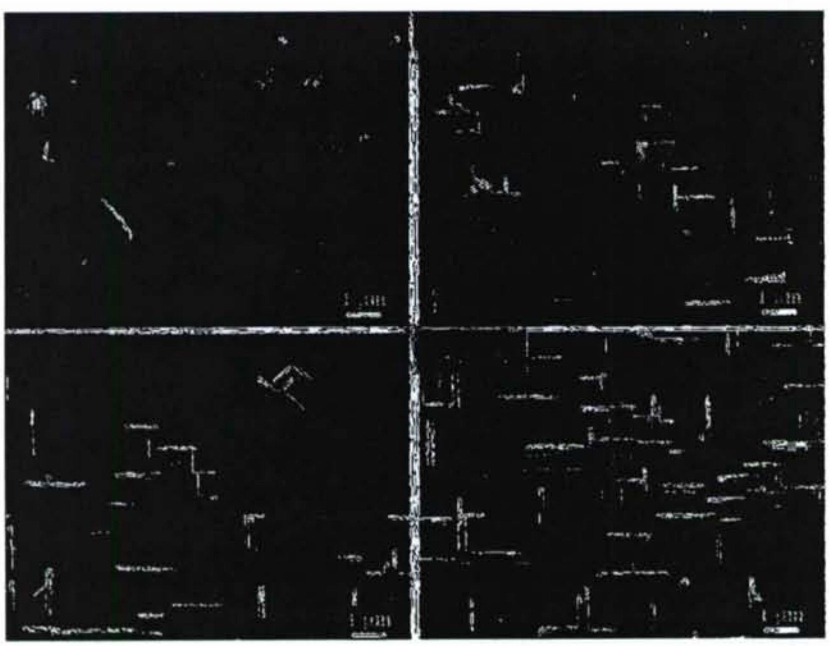

**Figure 8.** Surface SEM images of MOCVD films after 1 to 4 passes. Thickness of YBCO deposited in each pass is 0.7 mcrions.

Another potential limiting factor was found from compositional mapping of the cross section of our thick MOCVD films, the results from which are shown in Figure 9. Figure 9 shows Y, Sm, Ba, and Cu maps obtained from a 2.8 micron film. In the top half of the film, Y and Cu rich secondary phases could be seen which could be a limiting microstructural factor.

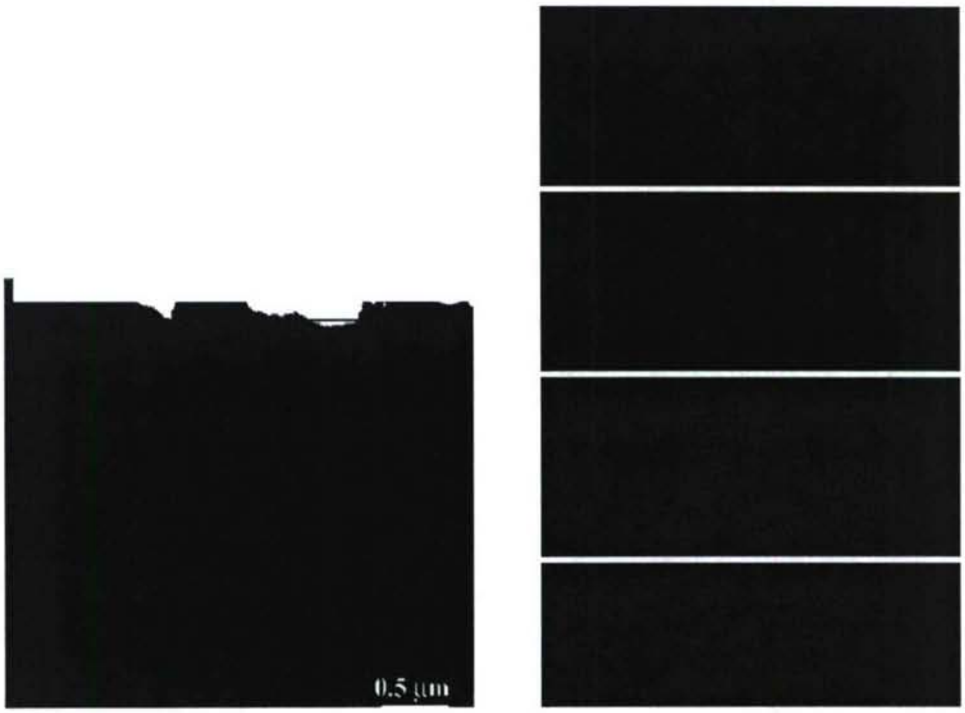


**Figure 9.** Cross section TEM image from a 2.8 micron thick HTS film grown by MOCVD and elemental mapping conducted in the area shown.

In summary, we achieved significant process in understanding the microstructural development of MOCVD-based 2G wires and in turn improved the critical current performance of thick films.

**Personnel Supported :**

Dr. Yimin Chen, Sr. Materials Scientist, MOCVD

Dr. Andrei Rar, Sr. Materials Scientist, Characterization

**Publications :**

1. Z. Chen, D. M. Feldmann, X. Song, S. I. Kim, A. Gurevich, J. L. Reeves, Y. Y. Xie, V. Selvamanickam and D. C. Larbalestier, "Three-dimensional vortex pinning by nano-precipitates in a Sm-doped YBa2Cu3O7-x coated conductor", *Supercond. Sci. Technol.* **20.** S205-S210 (2007)
2. V. Selvamanickam, Y. Chen, X. Xiong, Y. Xie, X. Zhang, Y. Qiao, J. Reeves, A. Rar, R. Schmidt, and K. Lenseth, "Progress in Scale-up of Second-generation HTS Conductor", *Physica C*, **463-465,** pp. 482-487 (2007)

**Interactions/Transitions :**

a. Conference Presentations :

1. ASC, Seattle, August 2006
2. ISS, Nagoya, October 2006
3. DOE Coated Conductor Workshop, Panama City, January 2007
4. MRS Spring Meeting, San Francisco, April 2007

b. Interaction :

As shown throughout this report, SuperPower has had extensive collaboration with LANL, FSU, and ORNL for understanding the in-field performance and to elucidate the microstructural reasons for the performance of our MOCVD-based conductors. Additional microstructural work, Raman Spectroscopy, and MOI was done together with ANL. Samples of MOCVD tapes have been provided to AFRL for various measurements. We have collaborated with Florida State University and University of Houston for stability, and mechanical property measurements. We have worked with Ohio State University on ac loss measurements and with California State University for VTLSM. SEM, TEM, AFM, and FIB analysis have been conducted by SuperPower staff and students supported by SuperPower at U. Albany.

c. Transition :

The ongoing AFOSR program will have a large impact on ongoing materials and device development programs at SuperPower. *The AFOSR program is a critical program* *at SuperPower for the development of YBCO 2G wire.* The success of the program will lead to a high-performance and potentially lower cost replacement for Bi-2223 conductor. Bi-2223 conductor is currently the main HTS conductor available in long lengths and is used in all demonstration projects. Based on its superior performance and potential lower cost, YBCO is the clear choice for HTS conductor for all these devices. *SuperPower recognizes this fact and is strongly supporting the AFOSR program through funds for capital equipment including MOCVD facilities.* Last year, SuperPower committed substantial funds towards capital equipment for MOCVD facilities that include upgrades to Pilot MOCVD facility and Prototype MOCVD facility, as well as purchase of a new Pilot MOCVD facility. The AFOSR program will eventually enable the fabrication of a high performance superconducting tape that can find wide use in military, electric power, magnetic, medical and applications.

**8. Inventions :**

None.

**9. Honors & Awards :**

None

**10. Research Planned for Following Year :**

This is the final year of the current AFOSR-funded program. This research will be carried over to a newly funded AFOSR program where we will continue our work on improving the critical current density of thick films in the range of 2 to 3 microns. Our goal is towards a 1000 A/cm conductor in a 2.5 micron thick HTS film by a process that can be readily scaled up to our Pilot-scale operation. We will continue our work on compositional modifications to enhance the in-field performance of our 0.7 micron and thick film tapes. We will also study the mechanical properties of the thicker film tapes.